CORROSION STUDIES ON 316 SS – B_4C COMPOSITE PREPARED BY POWDER METALLURGICAL ROUTE

A SYNOPSIS

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1. INTRODUCTION

The powder metallurgy process provides a host of advantages over competing metalworking technologies. These all add up to part-to-part uniformity for improved product quality, shape and material flexibility, application versatility, and cost effectiveness [1].

1.1 316 Stainless steel

316 Stainless steel (316SS) is a type of austenitic stainless-steel alloy that contains specific percentages of various elements to enhance its corrosion resistance, mechanical properties, and overall performance. 316SS exhibits excellent resistance to general corrosion in most environments, including exposure to atmospheric conditions, water (both fresh and saltwater). This resistance is due to the stable and protective oxide layer on its surface. Its ability to withstand various corrosive environments, resistance to acids and alkalis, and durability make it a reliable choice for industries and applications where corrosion protection is crucial. Table 1. shows that the chemical composition of 316SS, where the elements applied in corrosion applications.

Table 1 Chemical composition of 316SS

Elements	Percentage (%)
Chromium	18
Nickel	14
Molybdenum	2.5
Manganese	2
Carbon	0.08
Silicon	0.75
Phosphorous	0.045
Sulfur	0.03
Iron	Balance

316 Stainless steel is commonly used in corrosion-resistant applications due to its excellent corrosion resistance properties. Here are several reasons why 316SS is a preferred choice for such applications:

- **High Chromium Content:** 316SS contains around 16-18% chromium, which is a critical element that forms a passive oxide layer on the surface when exposed to oxygen. Chromium is the key element responsible for the corrosion resistance of stainless steel.

- **Nickel Content:** 316SS also contains 10-14% nickel, which contributes to its corrosion resistance. Nickel enhances the passivation process and helps maintain the stability of the oxide layer. It improves the steel's ability to withstand corrosion in a variety of environments.
- **Molybdenum Addition:** 316SS often includes 2-3% molybdenum, which enhances its resistance to pitting and crevice corrosion.
- **Versatility:** 316SS is versatile and can be used in various environments, including marine, coastal, industrial, and chemical settings.
- **Longevity:** 316SS has a long service life, which reduces maintenance and replacement costs. Its durability and resistance to corrosion contribute to its cost-effectiveness over time.

1.2 Boron carbide

Boron carbide (B_4C) is a ceramic compound composed of boron and carbon atoms. It is one of the hardest known materials, ranking just below diamond in terms of hardness. Boron carbide has a wide range of applications due to its exceptional properties, including:

- **Hardness:** Boron carbide is extremely hard, making it highly abrasion-resistant. Its hardness is one of the primary reasons for its use in various industrial and military applications.
- **Lightweight:** Despite its hardness, boron carbide is relatively lightweight compared to many other hard materials, such as tungsten carbide. This makes it valuable in applications where weight reduction is essential.
- **Neutron Absorption:** Boron carbide is an excellent neutron absorber, which is important in nuclear applications. It is used in control rods in nuclear reactors to regulate the rate of nuclear reactions and as a neutron shield to protect personnel and equipment from radiation.
- **Nuclear Industry:** Apart from neutron shielding and control rods, boron carbide is used in various applications within the nuclear industry, including as shielding material for radiation therapy and as a moderator in certain nuclear reactors.
- **Control of Nuclear Reactions:** In nuclear reactors, controlling the rate of nuclear reactions is essential to maintaining stable and safe operation. Boron carbide is used in the form of control rods or pellets within nuclear reactors.

Inserting the boron carbide control rods absorbs neutrons and slows down or stops the nuclear reaction, providing a reliable and controllable means of managing reactor power levels.

- **Nuclear Research:** In various nuclear research applications, scientists use boron carbide as a neutron absorber and moderator to conduct experiments and studies. It helps researchers control and manipulate neutron flux to study nuclear reactions and materials.

1.3 Spark plasma sintering

Spark Plasma Sintering (SPS), also known as Field Assisted Sintering Technique (FAST), is an advanced powder metallurgy process used for the densification and consolidation of materials, particularly ceramics, metals, and composites. It is a high-temperature, high-pressure sintering technique that offers several advantages over traditional sintering methods. The spark plasma effect rapidly heats the particle contacts, causing them to reach high temperatures within a very short time.

One of the key advantages of SPS is its relatively short processing time. It can sinter materials in a matter of minutes to hours, as compared to traditional sintering methods that may take much longer. SPS can be performed under controlled atmospheres, including vacuum or inert gas environments, which can help prevent unwanted chemical reactions or contamination during the sintering process. SPS can be applied to a wide range of materials, including ceramics, metals, composites, and even nanomaterials.

1.4 Importance of Spark Plasma Sintering Process

Applying pressure while at the same time applying (pulsed) current flow through the sample (in tungsten carbide or graphite die) at vacuum, which provides the heating effect and no atmospheric interaction on the samples.

- **High Densification:** SPS can produce materials with high density and improved mechanical properties.
- **Fine Microstructure:** It can yield materials with fine-grained microstructures, which can enhance material properties.
- **Rapid Sintering:** SPS is capable of achieving high densities in a short amount of time, which is beneficial for both efficiency and energy savings.

- **Uniform Heating:** The spark plasma effect provides uniform and controlled heating, reducing the risk of temperature gradients and distortion in the final product.

2. LITERATURE SURVEY

304 Stainless steel (304SS) is one of the promising materials used for these applications because of its superior corrosion resistance, high strength, toughness, and good weldability Baldev et al. [2]. However, this material suffers from insufficient wear resistance and radiation tolerance Balakrishnan et al. [3]. The shortcomings of this metal alloy can be resolved by the incorporation of hard ceramic reinforcements Saxena et al. [4]. Ceramic reinforcements into the metal matrix improve the properties such as density, hardness, and wear resistance of the matrix material without impacting corrosion resistance Martin et al. [5].

Among these ceramics, boron carbide (B_4C) is the third hardest material, with good chemical stability, high strength, and low density (2.52 g/cc). In addition, because of its ability to absorb neutrons, B_4C-reinforced composites can be effectively implemented for applications in nuclear industries. Aluminum–B_4C composites have gained immense interest in the last decade for the production of wear-resistant and neutron absorbent materials, but these materials sometimes suffer from seizure and galling at high-pressure working conditions Matovic et al. [6].

The B_4C-reinforced 304SS composite with neutron shielding properties wherein the B_4C content was optimized through the genetic algorithm program Suryanarayanan [7]. With the incorporation of spherical carbide particles, the density was reduced by 33% and a significant increase in radiation tolerance was achieved Shikalov et al. [8]. As mentioned earlier, the mechanism used for nuclear applications works under dynamic contact stress conditions, because of which, the SS–B_4C composites still demand to test tribological parameters such as wear and friction coefficient Sun et al. [9] and Akhtar et al. [10]. The properties of the composite depend upon the manufacturing method, reinforcing material, its fraction and distribution in the matrix, and interaction between the components Guo et al. [11] and Sahu et al. [12]. Conventional methods like stir casting, powder metallurgy (P/M), hot pressing, and extrusion along with some advanced

methods like spark plasma sintering and physical vapor deposition are available for the fabrication of ceramic-reinforced metal matrix composite (MMC) Malaki et al. [13] and Moriera et al. [14]. With better material utilization, simplicity in the incorporation of hard materials into the matrix and proper distribution of particulates into the matrix, P/M is a cost-effective technique to produce ceramic-reinforced metal composites Oke et al. [15]. On the other hand, porosity is induced in the composite structure during the sintering process which impacts the hardness of the material adversely Sharifi et al. [16] and Van Dong et al. [17].

The wear behaviour of the material in nuclear applications decides the maintenance cycle and overall life of the material in extreme conditions. Corrosion depends upon the type of material, its microstructure, roughness, porosity, and hardness. The hardness of any material affects the wear properties. The inclusion of a soft matrix with hard reinforcement increases the hardness along with the improvement of other properties such as density. An increase in the hardness of 304SS–B_4C composite up to 7.5%, with reinforcement of 3% was reported. Although the percentage of B_4C particles covered in ASTM A887 is only up to 0.25%, composites with ∼16 and ∼25% of B_4C reinforcement have been developed through PM and infiltration casting Ram Singh et al. [18]. Comprehensive studies on fabrication and characterization of SS–B_4C MMC have been carried out the tribological behavior of SS–B_4C composite. The current study is focused on the development and tribological testing of 304SS-B_4C composite material for high-speed low contact pressure nuclear applications. Composites with 0, 5, 10, and 15% B_4C volume reinforcement by PM method are produced Van Dong et al. [19].

304SS and 316SS are investigated that the atmospheric, sea water splash zone and underground environments to evaluate the corrosion rate for 14 months by adding TSP, chloride and sulphite in all weather conditions. Thus, the result shows that the 316SS has better corrosion resistance compared to 304SS (also satisfied in all conditions) Ul-Hamid et al. [20]. The EBSD is preferable for optical microscopy because it provides greater resolution and more accurate data for detailed microstructure characterization. EBSD is used for bulk materials rather than thin foils. From the samples, the data are obtained, e.g., near surface, breakages, and weld surface. Multiple grains and subgrains can be characterised by using a single map Oketola et al. [21]. Using SPS process, the result of EBSD research have revealed that higher sintering temperatures and improper

parameters leads to bigger grain sizes. It was observed in elevated sintering temperature samples Taylor et al. [22]. SS microstructure reported that the mechanism of different grain boundaries, bulk mechanisms, nuclei formation of sub grains, and merging processes occurs in powder metallurgy Mandal et al. [23].

3. RESEARCH OBJECTIVES

- To fabricate a metal matrix composite with 316 stainless steel as matrix and B_4C as reinforcement.
- The composite material will be prepared through the Spark plasma sintering process with different parameters such as temperature, pressure, heating rate and dwell time.
- The prepared composite will be subjected to electrochemical corrosion to investigate the corrosion resistance behavior of the fabricated composite.
- The optimum parameter for better corrosion resistance will be determined by optimization and will be examined by SEM images, EDAX mappings and EBSD analysis.
- The aim was to significantly improve the control rod 316SS – 10wt.% B_4C composite (such as less porosity, hardness and corrosion resistance) in order to avoid the material replacement.

4. RESEARCH PROBLEM

- The control rod of a light water nuclear reactor is constructed of a pole comprising stainless steel filled with a boron carbide (B_4C) core.
- A 1000 MW class contains about 1200 kg B_4C and about 17000 kg stainless steel 304 as a control rod component in the reactor core.
- The weights of these materials are equivalent to about 5% to 10% of the total core materials inventory, respectively.
- During accidents, the chemical interactions may corrode the control rod. Thus, an attempt is proposed to fabricate the control rod as a composite bar.
- Through powder metallurgy technique (SPS is chosen), the B_4C particles will be dispersed in stainless steel 316.

- This may prevent the chemical degradation at higher temperatures. Additionally, the corrosion resistance behavior of the prepared composite (316 SS–B_4C) will be investigated.

5. ORGANIZATION OF THESIS

Chapter 1: Introduction

This chapter is discussed about introduction of composites, powder metallurgy, 316 stainless steel, boron carbide, spark plasma sintering and the corrosion behaviour of the composites. The composites of austenitic stainless steel different grades are discussed and compared in metal matrix composites. The corrosion applications are discussed in SS. From nuclear applications research, B_4C is chosen as an additive for the corrosion studies. The mechanical testing and corrosion test involved in various powder metallurgical route was discussed.

Chapter 2: Literature Review

A detailed literature survey provided on the recent research regarding various issues including powder metallurgy, sintering process, mechanical testing and corrosion studies. Further this chapter is discussed about the microstructure analysis of fabricated sample and porosity. The 304 stainless steel was widely used in all applications including corrosion and nuclear applications studies. The spark plasma sintering method is an advance powder metallurgy method for the fabricating composites. Based on the literature review, it presented the research gap in this area. The objectives of this research work have been articulated as well.

Chapter 3: Materials and Experimental details

This chapter provides the research methodology used for the research is shown in Figure 1. 316 stainless steel is chosen as a base material and boron carbide is chosen as reinforcement of the composite (which is added 10wt.% to the base material). These composites are fabricated in spark plasma sintering method. After the material fabrication

the porosity, microstructure analysis like SEM, EBSD were analysed, micro-Vickers hardness test and Electrochemical corrosion test was investigated.

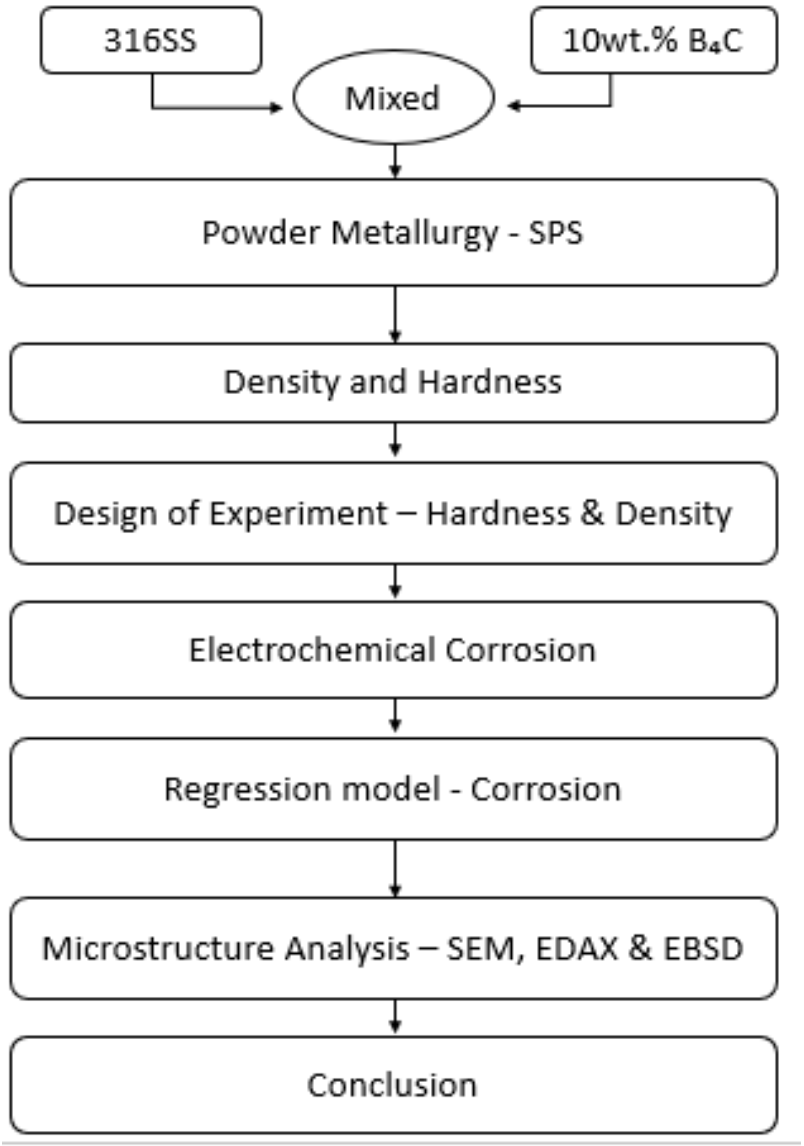

Figure 1 Research methodology

Chapter 4: Density and Hardness

This chapter describes the microstructure analysis of 316SS- 10wt.% B_4C composite prepared in spark plasma sintering method. The following methods are porosity, scanning electron microscopy (SEM), Energy dispersive X-ray analysis mapping (EDAX), and Electron backscattered diffraction (EBSD) are discussed.

4.1 Porosity

To determine the experimental density and to evaluate the porosity of 316SS-10 wt.% B_4C samples, dimensional and weight measurements are considered. According to Archimedes' principle, the weight of the fluid that the body disperses is equal to an upward buoyant force that is applied to a body submerged in a fluid, whether completely or partially. It is formulated by Archimedes where the porosity is measured using a specimen from the following equation. (1)

$$\% \text{ Porosity} = \left(1 - \frac{\text{Experimental Density}}{\text{Theoretical Density}}\right) \times 100 \quad (1)$$

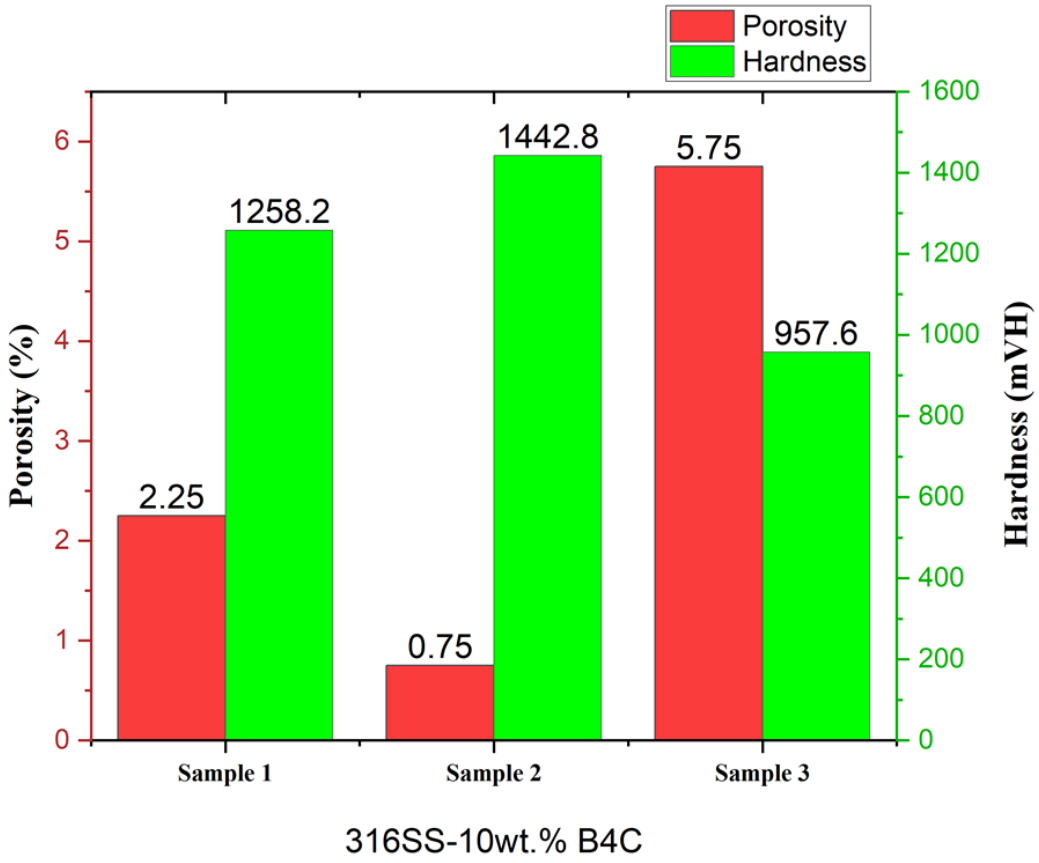

Figure 2 Graphical representation of porosity and hardness

4.2 Micro Vickers Hardness test

The Micro Vickers hardness (micro HV) was measured on the polished samples. Microhardness testing was performed on the sintered samples utilizing a 1-kg force and

a 10-s dwell duration. The ASTM E384-11 standard was used to assess the micro HV of the sample. The graphical representation of porosity and hardness of the sintered samples are shown in Figure 2. The best three sintered samples are chosen from the result and plotted. The sample 1 is sintered at 800°C, sample 2 is sintered at 900°C and sample 3 is sintered at 1000°C. From the graph, the sample 2 sintered at 900°C has low porosity and high hardness value compared to the sample 1 sintered at 800°C and sample 3 sintered at 1000°C.

Chapter 5. Design of Experiment

Taguchi technique is used to optimise the trials in SPS. The sintering temperatures (800, 900, and 1000 °C), pressures (60, 70, and 80 MPa), dwell times (5, 10, and 15 min), and heating rates (100, 200, and 300 °C/min) are selected as the factor variables for the experiment.

5.1 Statistical tool

The significance of the input parameters is also examined using the statistical technique known as ANOVA. The Taguchi L9 3^4 orthogonal array is used, giving the trial a total of nine runs.

The SPS parameters that are used to represent this composite material, this research uses statistical techniques from the Taguchi Design of Experiments (DOE) and the Analysis of Variance (ANOVA) statistical methodology. By using the sintering temperature, dwell time, pressure, and heating rate, this model is intended to generate 316SS-10 wt.% B_4C composite to forecast the density, microhardness and corrosion.

Chapter 6: Electrochemical Corrosion behaviour

This chapter includes the electrochemical corrosion behaviour of the 316SS – 10wt.% B_4C composite and SEM observations are discussed. The hardness test of the 316SS – 10wt.% B_4C composite are investigated and compared with the samples to find the best parameter.

6.1 Electrochemical corrosion

On the ACM corrosion monitoring electrochemical workstation, a standard two-electrode cell arrangement has been utilized for all electrochemical experiments, namely calomel electrode and platinum electrode. The potential discussed in this work has been evaluated with respect to the value of saturate calomel electrode (SCE). After establish a constant open-circuit potential (OCP), the samples are tested in 3.5 wt.% NaCl solution with distilled water (pH 7) at 25°C for 480 s. The electrochemical corrosion test was been performed. The experimental data and Tafel plot curve are analysed by the commercial software ACM Instruments (v5). The best corrosion parameters are compared and discussed.

6.2 Development of Mathematical model

The mathematical modelling can be calculated by two following methods, weight loss method and corrosion evaluation. The weight loss method is the pretreated specimen at initial stage weights were noted and were immersed in the experimental solution (corrosion test). After the experimental test, the final weights were noted. From the initial and final weight of the specimen the weight was calculated. (2)

$$CR = 534W/DAT\ mm/yr \qquad (2)$$

where W—Weight loss in milligrams (mg), D—Density in grams per cubic centimetre (g/cm³,) A—Area of the specimen (mm³) T – Time (sec).

Chapter 7: Comparison of Actual vs Predicted corrosion value using regression model.

In this chapter, the comparison of SS316-10wt.% B_4C composite of corrosion studies using regression model was analysed. The corrosion test was compared from the experimental value and predicted value. The corrosion error is 1% in which the validation is considered to be significant.

Chapter 8: Microstructural Analysis

In this chapter, the microstructural analysis of SS316-10wt.% B_4C composite was investigated and reported before and after corrosion.

8.1 SEM

The SEM are conducted in powder mixture to find the dispersion of B4C particles in SS316 and sintered sample. The EDAX was utilized to identify the chemical composition of SS316 and 10 wt.% B_4C composites. After corrosion the samples are analysed in order to find the porous and material damage.

8.2 EBSD

Using TSL-OIM (TexSEM laboratories- Orientation imaging microscopy) system, the EBSD data are collected through FEI Quanta 200 SEM operating at 20 KV. In each sample, an approximate area of 2mm x 2mm and a step size of 0.5 µm are covered by EBSD. For estimations of Grain orientation spread (GOS), Grain average misorientation (GAM) and Kernel average misorientation (KAM), first the grains were identified from the continuous presence of grain boundaries (less than 5° misorientation boundaries). When the misorientation exceeds 5° between two neighbouring orientation points, it is considered as the grain boundary. GAM is the misorientation between neighbouring measurement points in a grain.

Chapter 9: Conclusion

This chapter presents the summary of the electrochemical corrosion behaviour as the conclusion of the research work. In addition, this chapter highlighting future scope of work.

Statistical Tool

- The initial experiment and optimization of input parameters were carried out in the confirmation experiment. The best input parameter was determined at 900°C temperature, 70 MPa pressure, 10 min dwell time, and 200°C/min heating rate, with a relative density error of 1% and a microhardness error of 1.6 % which is considered as a significant result.

- The results obtained by regression equations closely correlate each other which validate the regression equations developed. A good result between the predicted and actual corrosion rate was observed.
- The results obtained from the statistical analysis are in good agreement with the experimental findings for the temperature, pressure, heating rate and time. It was found that corrosion rate increases with increasing temperature.
- The developed mathematical models can be used to predict the corrosion values in terms of corrosion control process parameters obtained from any combinations within the ranges studied.

Electrochemical corrosion

- The microstructure, mechanical and corrosion behavior of 316SS-10 wt.% B_4C composite samples 1, 2 and 3 have been systematically investigated.
- Long term annealing of sample 3 at 1000 °C results in abnormal crystal grain development and poor structural uniformity. When compared sample 1, sample 2 has the best corrosion rate, due to its moderately uniform dimensions and fewer grain boundaries.
- As a result, a reduction in pits implies an overall enhancement in the corrosion resistance of 316SS- 10wt.% B_4C materials.

Microstructural Analysis

- Using the SPS process, the samples are tested with different sintering temperatures, and the results of the EBSD research have revealed that higher sintering temperatures and improper process parameters lead to bigger grain sizes.
- The orientation maps show that the sample sintered at 900°C possesses <111> texturing. The present research has revealed that the samples that are sintered at the temperatures of 800°C and 1000 °C have very low relative frequencies of high-angle boundaries and they provide high grains without constant recrystallization.

Scope of Future Work

- The future work is analyzing various corrosion studies in the SS316-10wt.% B_4C composite.
- Further, simulation work can be done to confirm better corrosion resistance.

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LIST OF PUBLICATIONS

1. **K. Baranidharan**, S. Thirumalai Kumaran, M. Uthayakumar, and P. Parameswaran, "Comprehensive review of various corrosion behaviours on 316 stainless steel", Metallurgical and Materials Engineering 27 (2) (2021), 115-135. **(Scopus)**
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3. **K. Baranidharan**, S. Thirumalai Kumaran, M. Uthayakumar, P. Parameswaran, and D. Arvindha Babu, "EBSD analysis of spark plasma sintered SS316-B_4C composite", Micron 166 (2023), 103401. **(Impact Factor: 2.4)**
4. **K. Baranidharan**, S. Thirumalai Kumaran, M. Uthayakumar, and P. Parameswaran, "Electrochemical polarisation studies on spark plasma sintered SS316-B_4C composite", Surface Review and Letters (2023). **(Impact Factor: 1.1)**
5. **K. Baranidharan**, S. Thirumalai Kumaran, M. Uthayakumar, and P. Parameswaran, "Optimization modelling of spark plasma sintering parameters of SS316-B_4C composite", International Journal on Interactive Design and Manufacturing (IJIDeM) (2023), 1-13. **(Impact Factor: 2.2)**